UNIVERSITY OF
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POLYMER SCIENCE PROGRAM
Institute of Materials Science

March 2, 1995

Dr. L. T. Kabacoff
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Dear Larry:

Enclosed are the quarterly report for the period Oct.-Dec. 1994 on the Synthesis & Processing of Nanostructured Steels Project. Also enclosed is the announcement for the symposium on Nanostructured Materials at the ACS National Meeting in Chicago in August 1995 and ICAM IV in Cancun in Sept. 1995. If possible, I would like to request your participation.

If you have any questions please call.
Best regards.

Sincerely yours,
K. Gonsalves
Kenneth E. Gonsalves
Associate Professor

encl:
CC: 1) Admn. Grants Officer
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QUARTERLY REPORT FOR THE PERIOD 10/1/94 TO 12/31/94

SYNTHESIS AND PROCESSING OF NANOSTRUCTURED STEELS
{ONR N00014-94-1-0833}

Submitted to

OFFICE OF NAVAL RESEARCH
MATERIALS SCIENCE DIVISION
ARLINGTON VA

ATTN: DR. L. T. KABACOFF

SUBMITTED BY

K. GONSALVES

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CT 06269

MARCH 1, 1995

High Resolution Electron Microscopy of Nanometric Gold particles

The morphological characteristics of nanometric gold particles were obtained using HRTEM. The instrument for this purpose was a JEOL-4000SX HRTEM microscope with a point-to-point resolution of approximately 0.17 nm. The samples were prepared via dispersion of the nanometric particles in methanol with subsequent deposition on carbon coated copper grids. The TEM observation show that the particles are in the range of 7 to 10.5 nm, altough there are a few particles with sizes less than 5 nm. This is illustrated in Fig. 1, where the size distribution(diameter) is displayed. Fig. 2 shows the HRTEM of the small gold particles. All the particle display the characteristic lattice fringes of the common HREM images sometimes with atomic resolution. This figure also illustrates different types of morphologies and particle sizes. Some of the small gold particles have clear indications of faceting, as is clearly pointed by the arrows in the figure. The larger gold particles always display arrangements of twin boundaries in their structure as illustrated in Fig. 3. The smallest gold particles showed perfect crystalline structure. However, in some cases the presence of twin boundaries is evident. This is shown in Fig. 4. Sometimes the particles are arranged one on top of the other, therefore Moiré fringes in the HREM images are expected as is illustrated in Fig. 5. It is also interesting to point out that from these HREM images there is no clear evidence of the presence of defects such as stacking faults and small dislocations in the small crystallites.

In the previous report the synthesis of surface modified gold particles had been reported. These particles whose HRTEM results are discussed above, did not show agglomeration in solution even after six months. Moreover, the particles could be treated like "discrete chemical molecules" and could be stored in the solid state and regenerated in solution with no indication of agglomeration or flocculation after long periods.

The initial work on the synthesis and characterization of these "model" nanometric materials is complete and will be reported at the ACS National Meeting in Chicago in August 1995. A manuscript is also in preparation including some light scattering and optical measurements.

The results of these surface modified systems indicate that inorder to synthesize and retain the nanometric size, the iron based materials

need to be examined under similar conditions. This work is in progress.

Nitridation of Fe-based Nanometric Powders

In work directly related to the Fe-based nanometric powders, surface nitridation studies have been initiated.

Surface Nitridation: In a 250 ml flask dry decalin(dispersant/solvent), 7 g of iron pentacarbonyl were placed in a flow of argon. Under strong mechanical stirring, and in argon environment, the reaction mixture was heated to 80°C for 24 h and subsequently at 110oC for 1 h and finally at 150-160°C for another 12 h. Shiny black particles were observed at the end of this period. Into this stirred slurry, ammonia was continuously passed at 185°C for 5 h. The reaction vessel was cooled to ambient temperature while the passage of ammonia was maintained. The powders were filtered in a Schlenk filter funnel. The sample was stable and could be stored. On heating these samples to 400°C in an ammonia environment a black pyrophoric powder was obtained. This indicates that the as-synthesized powder is nitrided on the surface, but at 400°C it reverts back to the initial iron. These studies are being expanded to enable the powders to retain their integrity while being easier to handle during processing. The above studies have included initial XRD, XPS studies. Details will be included in the next report.

In addition, preliminary work on the synthesis of nanometric Fe-base powders via *ultrasonication* have also been initiated and in progress.

FIGURE CAPTIONS

Fig. 1. Particle size distribution of the nanometric gold particles. The size represents the largest of the diameter associated with the particles.

Fig. 2. HREM image of the gold nanoparticles. Some of the particles show clear signs of facetting (indicated by the arrows).

Fig. 3. HREM image of the gold nanoparticles with indications of twin boundaries in the large crystallites. (those pointed by the arrows).

Fig. 4. Small gold particle with twin boundaries indicated by the arrows.

Fig. 5. Moire fringes produced by the superposition of crystalline particles (indicated by small arrows), also a large particle with twin boundaries (large arrow).

fig. 1

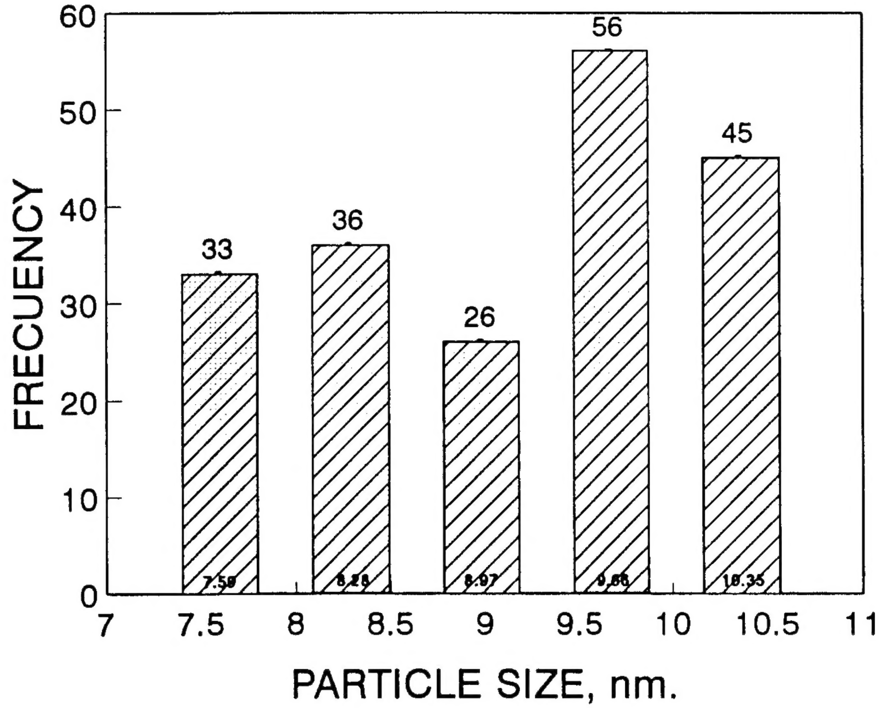
FRECUENCY
60
50
40
30
20
10
0
33
36
26
56
45
7.59
8.28
8.97
9.66
10.35
7
7.5
8
8.5
9
9.5
10
10.5
11
PARTICLE SIZE, nm.

FIG. 1

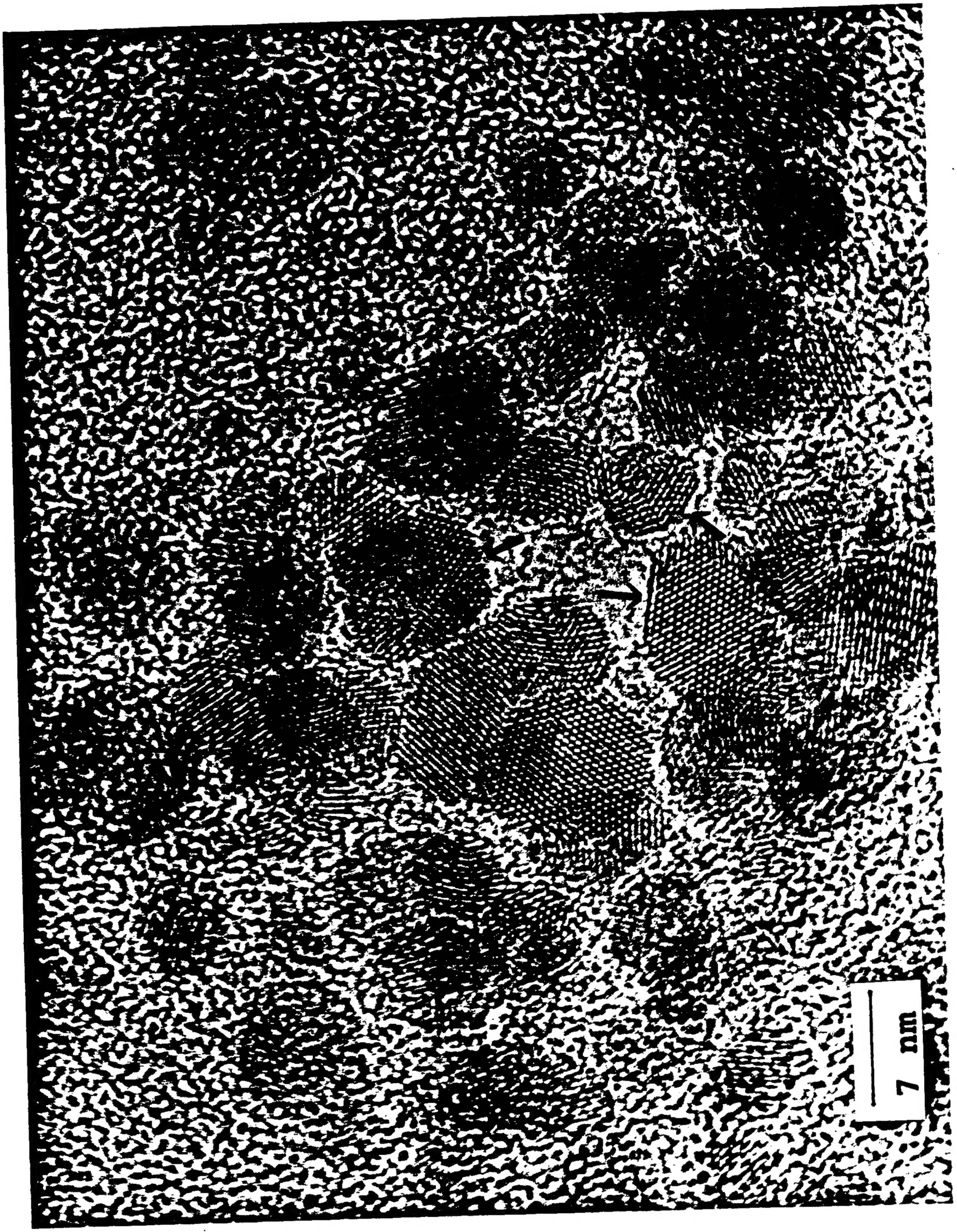

FIG.2

fig 3

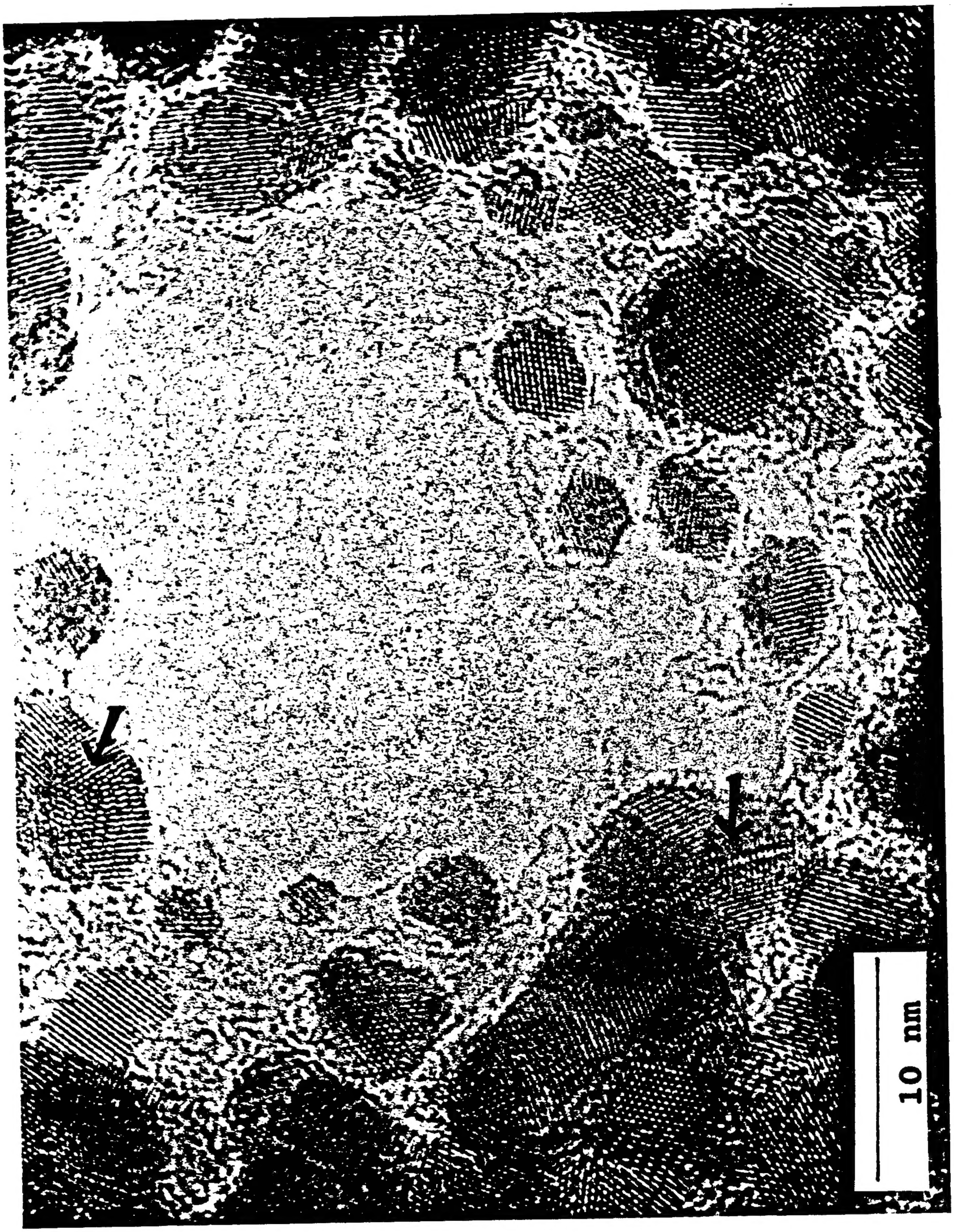

FIG. 3

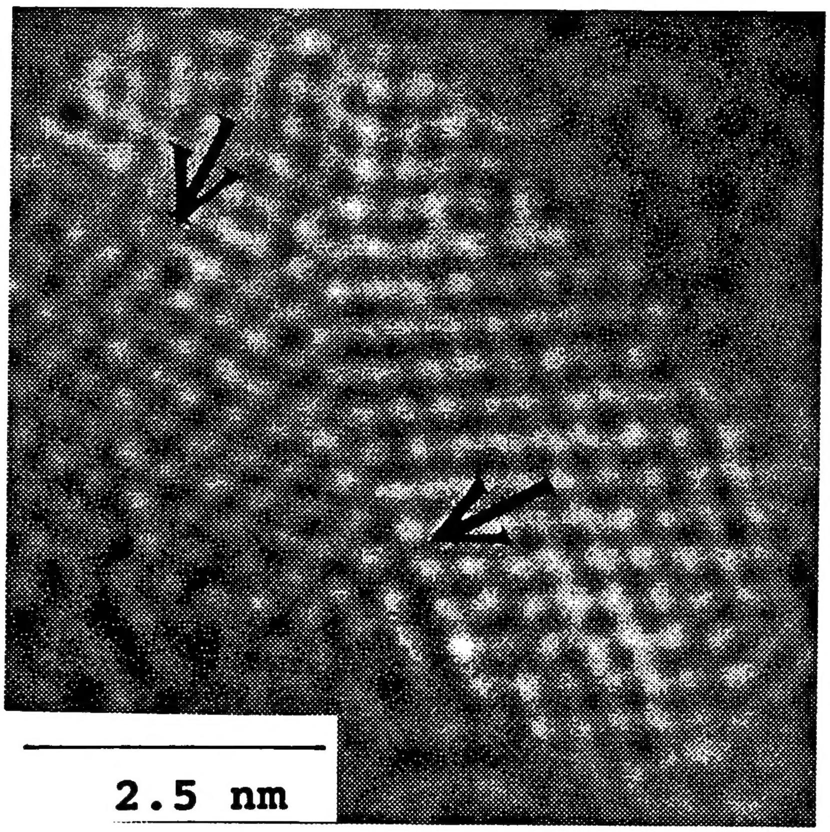

FIG. 4

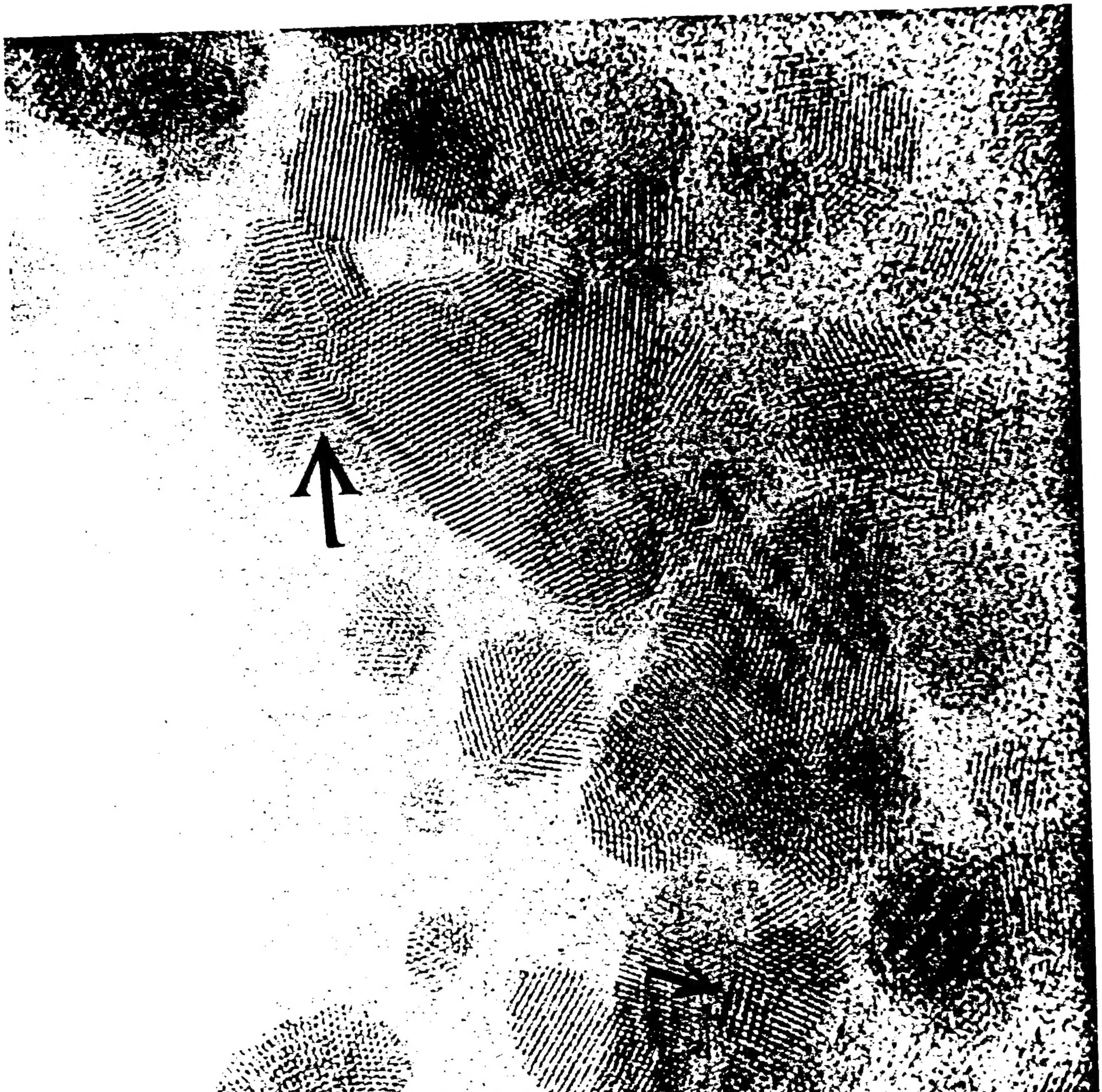

FIG. 5